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Indian Standard

METHODS OF CHEMICAL ANALYSIS OF BRONZES

PART 6 DETERMINATION OF ZINC BY COMPLEXOMETRIC (EDTA) METHOD

(First Revision)

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(First Revision)

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METHODS OF CHEMICAL ANALYSIS OF BRONZES

PART 6 DETERMINATION OF ZINC BY COMPLEXOMETRIC (EDTA) METHOD

(First Revision)

0. FOREWORD

0.1 This Indian Standard (Part 6) (First Revision) was adopted by the Bureau of Indian Standards on 22 July 1987, after the draft finalized by the Methods of Chemical Analysis of Non-Ferrous Metals Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 IS : 4027, first published in 1967, covered determination of copper, lead, tin, manganese, phosphorus, nickel, iron, silicon, aluminium, zinc and antimony in bronzes. While reviewing this standard, the Sectional Committee decided that it is convenient to revise this standard in series of parts which, on publication, supersedes the relevant method for determination given in IS : 4027 - 1967*. This part is one of that series and covers the determination of zinc by complexometric (EDTA) method. The other parts are as follows:

Part 1 Determination of copper and lead by electrolytic method

Part 2 Determination of manganese by photometric method

Part 3 Determination of phosphorus by volumetric method

Part 4 Determination of nickel by photometric method

Part 5 Determination of tin by iodimetric method

Methods for chemical analysis of other constituents in bronzes, namely, aluminium, iron, silicon and antimony are under preparation and will be published in subsequent parts of the above series.

0.3 In this revision, limitations of EDTA method for determination of zinc in bronzes has been prescribed.

*Methods of chemical analysis of bronzes.

0.4 The methods of analysis prescribed in this standard may primarily serve as referee methods and may also be used by the laboratories for their day-to-day work. Due consideration has been given in the preparation of this standard to the facilities available in the country for such analysis.

0.5 In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed of calculated, shall be rounded off in accordance with IS : 2 - 1960*.

1. SCOPE

1.1 This standard (Part 6) prescribes a method for determination of zinc in the range as specified in the relevant Indian Standards on bronzes.

NOTE — This method is not applicable when zinc is less than 0·2 percent.

2. SAMPLING

2.1 Samples shall be drawn and prepared in accordance with IS : 1817-1961*.

3. QUALITY OF REAGENTS

3.1 Unless specified otherwise, analytical grade reagents and distilled water (*see* IS : 1070 - 1977†) shall be employed in the test.

4. DETERMINATION OF ZINC BY COMPLEXOMETRIC (EDTA) METHOD

4.1 Outline of the Method — After removal of copper and lead, zinc is precipitated as sulphide, filtered, dissolved in sulphuric acid titrated with EDTA solution.

4.2 Reagents

4.2.1 *Dilute Nitric Acid* — 1 : 1 (*v/v*).

4.2.2 *Tartaric Acid Solution* — 30 percent (*w/v*).

4.2.3 *Concentrated Ammonium Hydroxide* — 20 percent.

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*Rules for rounding off numerical values (*revised*).

†Methods of sampling non-ferrous metals for chemical analysis.

‡Specification for water for general laboratory use (*second revision*).

4.2.4 *Methyl Red Indicator Solution* — 0·1 percent (*w/v*). Dissolve 0·1 g of methyl red in 3·72 ml of sodium hydroxide solution (0·1 N) and dilute to 250 ml with water. Filter, if necessary.

4.2.5 *Formic Acid Mixture* — To 20 ml of formic acid (*rd* = 1·20), add 25 g of ammonium sulphate and 3 ml of ammonium hydroxide (20 percent), and dilute to 100 ml.

4.2.6 *Hydrogen Sulphide* — gas.

4.2.7 *Formic Acid Wash Solution* — Dilute 4 ml of formic acid (*rd* = 1·20) to one litre with water.

4.2.8 *Dilute Sulphuric Acid* — 1 : 1 (*v/v*).

4.2.9 *Sodium Hydroxide Solution* — 200 g/litre.

4.2.10 *Buffer Solution* — Dissolve 54 g of ammonium chloride in 300 ml of water, add 350 ml of ammonium hydroxide (20 percent) and dilute to one litre. This solution has a *p*H of 10.

4.2.11 *Eriochrome Black-T Indicator Solution* — Dissolve 0·4 g of the sodium salt of eriochrome black-T in 20 ml of ethanol, add 30 ml of tri-ethanolamine and store in polythene dropping bottle.

4.2.12 *Standard EDTA Solution (0·05 M)* — Dissolve 18·6 g of the salt in 600 ml of hot water. Cool to room temperature and dilute to one litre with water. Standardize the solution as given in **4.2.12.1.**

4.2.12.1 Transfer to 800-ml beaker an aliquot of the standard zinc solution (*see* **4.2.13**) approximately equal in zinc content to the aliquot of the sample. Continue as directed in **4.3.4.** Calculate the equivalent of the EDTA solution in terms of g of zinc per ml of solution.

4.2.13 *Standard Zinc Solution* — Dissolve 4·000 g of pure electrolytic zinc in 200 ml of dilute hydrochloric acid (1 : 4), cool and make up to one litre.

4.3 Procedure

4.3.1 Dissolve 1·000 g of sample in dilute nitric acid and remove copper, lead and tin [*see* IS : 4027 (Part 1)-1987*].

4.3.2 Add 25 ml of tartaric acid solution and neutralize with concentrated ammonium hydroxide using methyl red as indicator. Add 25 ml of formic acid mixture, heat to 70 - 80°C. Add a little paper pulp and

*Methods of chemical analysis of bronzes : Part 1 Determination of copper and lead by electrolytic method.

pass hydrogen sulphide gas rapidly through the solution for 30 minutes. Allow the precipitate of zinc sulphide to coagulate, filtre on a pulp pad and wash with warm formic acid wash solution.

4.3.3 Dissolve the precipitate of zinc sulphide in hot dilute sulphuric acid, boil to expel all hydrogen sulphide gas. Cool and make up to 250 ml.

4.3.4 Take a suitable aliquot. Neutralize the solution with sodium hydroxide solution using methyl red indicator. Add 30 ml of buffer solution, 5 drops of erichrome black - T indicator and titrate slowly with EDTA to bluish green end point.

4.4 Calculation

$$\text{Zinc, percent} = \frac{A \times B}{C \times 10}$$

where

A = volume in ml of EDTA solution required for titration of the solution,

B = zinc equivalent in g/l of EDTA solution, and

C = mass in g of sample represented by the aliquot.

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